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(54) Treatment of food products and by-products.

(57) The present invention is concerned with a process for treating food products and by-products to remove therefrom off-flavours, off-colours, off-smells and undesirable impurities. The process comprises treating the food products and by-products, for example dairy products, soya products and by-products thereof, with adsorbent resin.

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The present invention is concerned with the treatment of food products and by-products. More particularly, the present invention is concerned with the treatment of food products and by-products to remove therefrom off-colours, off-flavours, off-smells and/or undesirable contaminants.

Food products and by-products are often subjected to stabilisation or sterilisation procedures during their processing. Such stabilisation or sterilisation procedures may, for example, involve subjecting the food products or by-products to elevated temperatures, high pressures, UV light, ultrasonics or x-rays. These stabilisation or sterilisation techniques may lead to the production of off-flavours, off-colours and/or off-smells in the food products and by-products.

For example, dairy products and by-products, e.g. milk and products made therefrom, are often subjected to treatment, prior to storage and transportation, to stabilise such products and by-products against microbiological actions and enzyme activities (known as enzyme inactivation or stabilization). This stabilisation is normally achieved by a heat treatment process such as pasteurisation, sterilisation or UHT (Ultra Heat Treatment - sometimes known as Ultra High Temperature). However, such heat treatment processes often result in the formation of off-flavours (off tastes), off-colours and/or off-smells in the dairy products and by-products which inhibit their acceptability by the end consumer. In this connection, as dairy products generally have subtle flavours and odours, the presence of off-flavours and off-smells in such dairy products becomes especially noticeable to the consumer. Similarly, dairy products generally have a light colour and this makes the presence of off-colours in such products espceially noticeable to the consumer.

Off-flavours, off-colours and/or off-smells in food products and by-products, for example dairy products and by-products, can also occur due to oxidation and microbiological spoilage of the food product or by-product. Further, in the case of dairy products and by-products, off-flavours, off-colours and/or off-smells can occur due to the use of enzymes (e.g. hydrolysation of milk and milk by-products). food products and by-products can also contain natural off-flavours, off-colours and/or off-smells, i.e. originating from the food products themselves. For example, in the case of milk products and by-products, off-smells can originate from the source of the milk, i.e. the cow, goat or sheep itself.

Clearly, it would be desirable if the off-flavours (off tastes), off-colours and/or off-smells often associated with food products and by-products such as dairy products and by-products, for example off-flavours, off-colours and/or off-smells resulting from heat treatment processes or other stabilisation/sterilisation steps, could be removed from, or at least minimised in, such products and by-products.

We have now found a process for the treatment of food products and by-products, preferably those food products and by-products that have been subjected to a heat treatment step or other stabilisation/sterilization step, which enables off-colours and/or off-flavours (off-tastes) and/or off-smells, for example off-flavours, off-colours and/or off-smells generated during processing of such products and by-products, to be removed, or at least minimised. The process also enables undesirable contaminants, for example residues of agricultural chemicals used in farming practises, e.g. pesticides, herbicides, fertilisers or veterinary preparations such as antibiotics and hormones, to be removed from, or at least minimised in, the food products and by-products. The process further enables the cloud stability of food products and by-products to be improved.

According to the present invention there is provided a process for the treatment of food products and by-products, which comprises contacting the food products or by-products with adsorbent resin.

The present invention also provides the use of adsorbent resin in the treatment of food products and by-products to remove off-flavours, off-colours and/or off smells from the food products and by-products.

As used herein, the term "food products and by-products" and "dairy products and by-products" are intended to mean "food products and by-products of such food products" and "dairy products and by-products of such dairy products", respectively.

Preferably, the food products and by-products treated in accordance with the process of the present invention are selected from dairy products, soya products, and by-products thereof, and are preferably selected from dairy products and soya products. In one embodiment of the present invention the food products and by-products are milk products or by-products, preferably milk products . In another embodiment of the present invention the food product or by-product is soya milk.

The food products and by-products may, for example, be in the form of liquid products, concentrated products or powdered products. Further, the food products and by-products may, for example, be in the form of blends, mixtures or reconstituted products. In one embodiment of the present invention, the food products and by-products may be split up into different phases before treatment in accordance with the present invention, one or more of the phases may then be subjected to treatment in accordance with the present invention, and the separate phases may then be reconstituted, i.e. the reconstituted product is formed after treatment in accordance with the present invention. An example of this is milk which may be separated into skimmed milk, cream, etc., one or more of the separate phases may then be subjected to treatment in accordance with the present invention, and the separate phases may then be reconstituted after treatment. Of course, it is also pos-

sible for the reconstituted product to be formed before treatment in accordance with the present invention in which case the reconstituted product is subjected to treatment with the adsorbent resin.

If necessary, the food product or by-product can be diluted with an appropriate diluent prior to its treatment in accordance with the present invention. For example, if necessary, the food product or by-product can be diluted with an appropriate diluent in order that it should be in a fluid form suitable for treatment with the adsorbent resin, for example, in order for it to have a viscosity sufficient to enable it to be treated with the adsorbent resin. For example, if the food product or by-product is in the form of a powder, it will be necessary to use a diluent in order for it to be in a form suitable for treatment with the adsorbent resin. If the food product or by-product is in the form of a concentrate, then it may be advisable or necessary to use a diluent, for example, so that it has a viscosity suitable for it to be treated with the adsorbent resin. The diluent may, for example, be a solvent or suspending agent for the food product or by-product, and a preferred diluent is water.

Examples of suitable dairy products and by-products include milk and products derived therefrom. The milk may, for example, be cow's milk, sheep's milk, goat's milk, camel's milk or mare's milk. Further, the milk may, for example, be in the form of whole milk, partially-skimmed milk, or fully skimmed milk. Examples of products derived from milk include whey, cream, hydrolysates, cheese and cheeselike products, yoghurt, tofu, koumiss, kefir and tätte. Products derived from dairy products and by-products may, for example, be in the form of blends, mixtures and reconstituted products.

Other, non-dairy food products which may be treated in accordance with the process of the present invention include soya products and by-products, for example, the so-called "soya milk" which has a milk-like appearance and may be used as a milk substitute, concentrated soya products and other such products. As in the case of dairy products, the soya products and by-products may, for example, be in the form of liquid products, concentrated products or powdered products. Further, as in the case of dairy products and by-products, the soya products and by-products may, for example, be in the form of blends, mixtures or reconstituted products. An example of a product derived from soya, and which can be treated in accordance with the process of the present invention, is oil obtained from the so-called soya milk.

The process of the present invention may, for example, be used to treat food products and by-products, preferably dairy products, soya products and by-products thereof, which have been subjected to a heat treatment process or other stabilisation process such as high pressure stabilisation, or stabilisation using UV light, ultrasonics or x-rays. In the case of dairy products and by-products, this heat treatment process may, for example, be pasteurisation, sterilisation or UHT.

It has been found that the use, in accordance with the present invention, of an adsorbent resin can remove, from the food products and by-products treated, trace impurities which give rise to the off-flavours (off-tastes), off-colours and/or off-smells, for example the off-flavours (off-tastes), off-colours and/or off-smells often present after heat treatment or other stabilisation processes. Also, it has been found that the use of the adsorbent resin does not adversely affect the nutritional value or the ionic composition of the food products and by-products. For example, it has surprisingly been found that, although milk and soya products are complex colloidal suspensions of proteins such products can be treated in accordance with the present invention without resulting in denaturing of the proteins or destruction of the colloidal suspension.

As the adsorbent resins are to be used, in accordance with the process of the present invention, for treating food products and by-products, it is important that they be of a quality suitable for use in food treatment, and it is also important that they be approved, where necessary, by the relevant authorities for such use. Also, the adsorbent resins should be physically and chemically stable to the operating conditions normally experienced in the food industry, for example the dairy industry.

Adsorbent resins, which are used in the process of the present invention have the advantage that they can be readily sterilised by chemical or physical means, for example, by the use of a high temperature, a feature which is very desirable in a material which is to be contactable with food products and by-products.

The adsorbent resins suitable for use in the present invention may, for example have ion exchange functionality. However, it is preferred that the adsorbent resins have no, or only limited, ion exchange functionaltiy.

Adsorbent resins suitable for use in the process of the present invention are typically macroporous resins, for example, macroporous resins having an average surface area of about 100 m^2/g or greater, preferably about 250 m^2/g or greater. The adsorbent resins may be polymeric adsorbents and carbonaceous adsorbents, and are preferably polymeric adsorbents. Suitable polymeric adsorbents include those based on crosslinked polymers formed from monomers comprising aromatic monomers, aliphatic monomers or mixtures thereof. Suitable aromatic monomers include phenol, styrene, and alkyl-substituted styrenes such as (α-methylstyrene, ethylvinylbenzene, p-methylstyrene and vinylxylene, and suitable aliphatic monomers include acrylic esters, methacrylic esters and acrylonitrile. Preferably, the polymers are crosslinked with polyethylenically unsaturated monomers, for example: aromatic crosslinkers such as divinylbenzene, divinyltoluene, trivinylbenzene, divinylchlorobenzene, diallylphthalate, divinylnaphthalene, divinylxylene, divinylethylbenzene, trivinylnaphthalene

and polyvinylanthracenes; aliphatic crosslinkers having a plurality of non-conjugated vinyl groups such as di- and polyacrylates and methacrylates, e.g. trimethylolpropane trimethacrylate, ethylene glycol dimethacrylate, ethylene glycol diacrylate, neopentyl glycol dimethacrylate, pentaerythritol tetra- and trimethacrylates and allyl acrylate; divinylaliphatic crosslinking monomers such as divinyl ketone and diethylene glycol divinyl ether; diacrylamides and dimethacrylamides such as N', N'-methylenediacrylamide, N',N'-methylenedimethacrylamide, and N',N'-ethylenediacrylamide; polyallyl aliphatic crosslinkers such as diallyl maleate, diallyl fumarate, diallyl succinate, diallyl carbonate, diallyl malonate, diallyl oxylate, diallyl adipate, diallyl sebacate, diallyl tartrate, diallyl tricarballylate, triallyl aconitate, and triallyl citrate; and the polyallyl and polyvinyl ethers of glycol, glycerol and pentaerythritol. Preferably, the adsorbent resin is formed from monomers comprising divinylbenzene and optionally other aromatic monomer such as styrene or ethylvinylbenzene. More preferably the adsorbent resin is formed from styrene and divinylbenzene. Levels of the crosslinking monomer are preferably about 25 weight percent or greater, and, where divinylbenzene is used, a preferred embodiment of the present invention uses adsorbent resins having levels of divinylbenzene of about 50 weight percent or greater. In one embodiment of the present invention the adsorbent is a polyvinylpolypyrrolidone or polyamide. The adsorbent resins may, for example, be non-ionic or have low ionic character such that they result in only limited changes in the ionic character of the treated food products and by-products.

Suitable carbonaceous adsorbents include partially pyrloyzed materials, for example, having an average surface area of about 100 m^2/g or greater, preferably about 250 m^2/g or greater. Such materials include activated carbon, such as activated charcoal from wood, bone, coal, lampblack and the like; and the more preferred carbonaceous adsorbents made by partial pyrolysis of macroporous, synthetic copolymers and ion exchange resins in the presence of a carbon fixing moiety, for example, a sulphonic acid group. Suitable monomers from which the synthetic copolymers and ion exchange resins may be made include those listed above as suitable for making the polymeric adsorbent resins. These more preferred carbonaceous adsorbents are typically pyrolyzed at temperatures of from about 300°C to about 900°C, preferably 300°C to 700°C, in a non-oxidising atmosphere, and the most preferred of these adsorbents has a carbon-to-hydrogen ratio of from 1.5:1 to 20:1 and at least 85% by weight of carbon.

Examples of suitable commercially available polymeric adsorbent resins are Amberlite XAD-4, Amberlite XAD-16, Amberlite XAD-7 and Duolite XAD-761, each of which is manufactured by Rohm and Haas Company, Philadelphia, United States of America. Examples of suitable carbonaceous adsorbents include activated carbon and the Ambersorb adsorbents manufactured by Rohm and Haas Company, Philadelphia, United States of America, e.g. Ambersorb 563.

The adsorbent resins, which preferably have a long useful life, may be regenerated, for example, by the use of regenerants normally available in the food industry, e.g. the dairy industry. Suitable regenerants include solvents, elevated temperatures, acidic regenerants, basic regenerants and oxidising regenerants, and combinations thereof. Examples of suitable solvents that may be used to regenerate the adsorbent resin include ethanol, methanol, propanol and isopropanol. In one embodiment of the present invention the regenerant comprises regenerant selected from sodium hydroxide, hydrochloric acid, nitric acid, sodium hypochlorite, hydrogen peroxide, peracetic acid, potassium hydroxide, phosphoric acid, steam, and hot water, and preferably comprises regenerant selected from sodium hydroxide, hydrochloric acid, nitric acid, sodium hypochlorite, steam, and hot water. The regeneration of the adsorbent resins may be carried out at elevated temperatures. When regenerated using elevated temperatures, the temperature may, for example, be at least 45°C, preferably at least 90°C, and more preferably at least 120°C. When regenerated using elevated temperatures, the temperature is preferably not more than 180°C.

In the process of the present invention, the food products and by-products may, for example, be contacted with the adsorbent resin by passing such product or by-product through a bed of the adsorbent resin. Alternatively, the adsorbent resin may be contacted with the food product or by-product by admixing the adsorbent resin with the food product or by-product, for example, in a batch contacting system or in a fluidised bed system or in such other mechanical contacting device normally used in the ion exchange industry.

In one embodiment of the process of the present invention, the food products and by-products are subjected to filtration, for example, ultrafiltration, microfiltration, hyperfiltration, reverse osmosis and/or centrifugation, e.g. high speed centrifugation or ultracentrifugation, prior to or after, preferably prior to, the treatment with the adsorbent resin.

By practice of the present invention it is possible to improve the quality of food products and by-products in one or more of the following respects:-

1. Flavour improvement by removing, or minimising, off-flavours (off-tastes).
2. Colour improvement by removing, or minimising, off-colours.
3. Improvement in odour by removing, or minimising, off-smells.
4. Improvement in purity by removing, or minimising, undesirable contaminants such as residues of agri-

cultural chemicals.

5. Cloud stability improvement, especially when the food product treated is the so-called "soya milk".

The following Examples are presented to illustrate certain embodiments of the present invention.

Examples

Examples 1 to 8:

A number of milk products and by-products from different sources such as cow, goat, sheep, and mare, as whole or skimmed product, were subjected to treatment with an adsorbent resin in accordance with the process of the present invention. A sample of soya milk was also subjected to treatment with an adsorbent in accordance with the process of the present invention.

The products tested were either fresh or had been subjected to heat treatment prior to being treated with the adsorbent resin.

The adsorbent resin used in the Examples was Amberlite XAD-16 and was added to the milk/soya products in an amount of 50g adsorbent resin/litre of milk/soya product. After addition of the adsorbent resin, the resulting mixture was shaken and then filtered to separate the resin from the milk/soya product. The treatment of the milk/soya product with the adsorbent resin was effected at room temperature (approx. 25°C).

The products were evaluated, both prior to and after treatment with the adsorbent resin, by a taste panel compose of eight people. The products were assessed based on the following criteria:-

1. Flavour (taste);
2. Bitterness;
3. Colour;
4. Smell (in the case of milk);
5. Mouth feel (in the case of soya milk); and
6. Cloud stability (in the case of soya milk).

For each of the assessements the product was assessed on a scale of 1 to 5, with 1 being the worst rating (i.e. poorest quality in respect of the particular criterium) and 5 being the best rating (i.e. the best quality in respect of the particular criterium).

The results obtained are summarised below.

Example 1:

Whole cow's milk, ca. 3.5% fat, fresh.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	4	2
After Treatment:	5	5	4	5

Example 2:

Whole cow's milk, ca 3.5% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

Example 3:

Whole cow's milk, ca 3.5% fat, pasteurised.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	4	4	3	4
After Treatment:	5	5	4	5

Example 4:

Whole goat's milk, ca 3% fat, fresh.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	4	2
After Treatment:	5	5	4	5

Example 5:

Whole goat's milk, ca 3% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

Example 6:

Skimmed cow's milk, less than 0.5% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

Example 7:

Cream from cow's milk, greater than 25% fat, pasteurised.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	3	2
After Treatment:	5	5	4	4

Example 8:

Whole soya milk, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	3	2
After Treatment:	3	3	4	4

	Mouth feel	Cloud stability
Prior to Treatment:	2	1
After Treatment:	4	5

Examples 9 to 11:

The procedure of Examples 1 to 8 was repeated on a number of milk and soya milk products except that the adsorbent resin used was Amberlite XAD-7. The results obtained are summarised below.

Example 9:

Whole cow's milk, ca. 3.5% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

Example 10:

Whole soya milk, Heat treated.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	3	2
After Treatment:	3	3	4	4

	Mouth Feel	Cloud Stability
Prior to Treatment:	2	2
After Treatment:	3	5

Example 11:

Whole sheep's milk, ca. 3% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	2	2
After Treatment:	5	5	4	4

Examples 12 to 14:

The procedure of Examples 1 to 8 was repeated on a number of milk and soya milk products except that the adsorbent resin used was Ambersorb 563. The results obtained are summarised below.

Example 12:

Whole cow's milk, ca. 3.5% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

Example 13:

Whole soya milk, heat treated.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	1	1	3	2
After Treatment:	4	4	4	4

	Mouth Feel	Cloud Stability
Prior to Treatment:	2	2
After Treatment:	4	4

Example 14:

Skimmed cow's milk, <0.5% fat, UHT.

	Flavour	Smell	Bitterness	Colour
Prior to Treatment:	2	2	1	2
After Treatment:	5	5	4	5

As can be seen from the above data, the quality of the products treated is improved by subjecting the products to treatment with the adsorbent resin.
"Amberlite" and "Duolite" are trademarks of Rohm and Haas Company, Philadelphia, United States of America.

Claims

1. A process for treating food products and by-products, which comprises contacting the food product or by-product with adsorbent resin.

2. A process as claimed in claim 1, wherein the adsorbent resin

3. A process as claimed in claim 2, wherein the adsorbent resin is a macroporous resin having a surface area of 100 m^2/g or greater, preferably 250 m^2/g or greater.

4. A process as claimed in any preceding claim, wherein the polymeric adsorbent resin is a crosslinked poly-

mer formed from monomers comprising styrene and divinylbenzene.

5. A process as claimed in any preceding claim, wherein the food product or by-product is passed through a bed of the adsorbent resin.

6. A process as claimed in any preceding claim, wherein the food product or by-product is subjected to filtration and/or centrifugation before or after, preferably before, treatment with the adsorbent resin.

7. A process as claimed in claim 6, wherein the filtration is ultrafiltration, microfiltration, hyperfiltration, or reverse osmosis, preferably ultrafiltration.

8. A process as claimed in claim 6, wherein the centrifugation is high speed centrifugation or ultracentrifugation.

9. A process as claimed in any preceding claim, wherein, subsequent to contact with the food product or by-product, the adsorbent resin is regenerated using solvent, elevated temperature, acidic regenerant, basic regenerant, oxidising regenerant, or a combination thereof.

10. A process as claimed in claim 9, wherein the regenerant comprises regenerant selected from sodium hydroxide, hydrochloric acid, nitric acid, sodium hypochlorite, hydrogen peroxide, peracetic acid, potassium hydroxide, phosphoric acid, steam and hot water, and preferably the regenerant comprises regenerant selected from sodium hydroxide, hydrochloric acid, nitric acid, sodium hypochlorite, steam and hot water.

11. A process as claimed in claim 9 or claim 10, wherein the regenerant comprises solvent selected from ethanol, methanol, propanol and isopropanol.

12. A process as claimed in any of claims 9 to 11, wherein the regeneration is carried out at a temperature of at least 45°C, preferably at least 120°C.

13. A process as claimed in any of claims 9 to 12, wherein the regeneration is carried out at a temperature of up to 180°C.

14. A process as claimed in any preceding claim, wherein the food product or by-product is selected from dairy products, soya products, and by-products thereof.

15. A process as claimed in claim 14, wherein the food product or by-product is a milk product or by-product thereof.

16. A process as claimed in claim 14, wherein the food product or by-product is soya milk.

17. Use of adsorbent resin in the treatment of food products and by-products to remove off-flavours, off-colours and/or off smells from the food products and by-products.

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EUROPEAN SEARCH REPORT

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
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Y	* abstract*	2,3	
Y	DATABASE WPI Week 9210, Derwent Publications Ltd., London, GB; AN 92-075411 & JP-A-4 018 436 (MITSUBISHI KASEI CORP) * abstract *	2,3	
X	PATENT ABSTRACTS OF JAPAN vol. 012, no. 037 (C-473)4 February 1988 & JP-A-62 186 742 (KURITA SEIBI KK) * abstract *	1,4,5,9, 10,14,15	
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	07 SEPTEMBER 1993	VAN AMSTERDAM L.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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EUROPEAN SEARCH REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
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X	PATENT ABSTRACTS OF JAPAN vol. 006, no. 202 (C-129)13 October 1982 & JP-A-57 110 166 (HIGETA SHIYOUYU KK) * abstract *	1,14,16	
X	DATABASE WPI Week 7342, Derwent Publications Ltd., London, GB; AN 73-63570U & JP-B-48 033 378 (KIKKOMAN SYOYU CO LTD) * abstract *	1,14,16	
X	DATABASE WPI Week 8444, Derwent Publications Ltd., London, GB; AN 84-273707 & JP-A-59 169 464 (HASEGAWA KK) * abstract *	1,9,14, 15	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
X	US-A-4 276 179 (J.W. SOEHNGEN) * claims 1-12 *	1,14,15	

The present search report has been drawn up for all claims

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BERLIN	07 SEPTEMBER 1993	VAN AMSTERDAM L.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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